

EFFECT OF COPPER ON THE AUSTEMPERING BEHAVIOUR OF SHPEROIDAL GRAPHITE IRON

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENT FOR THE DEGREE OF

**Bachelor of Technology
in
Metallurgical and Materials Engineering**

**By
KAUSIK TAMULI (107MM003)
&
AYAZ KHAN (107MM005)**



**Department of Metallurgical and Materials Engineering
National Institute of Technology
Rourkela
2011**

EFFECT OF COPPER ON THE AUSTEMPERING BEHAVIOUR OF SHPEROIDAL GRAPHITE IRON

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENT FOR THE DEGREE OF

**Bachelor of Technology
in
Metallurgical and Materials Engineering**

**By
KAUSIK TAMULI (107MM003)
&
AYAZ KHAN (107MM005)**

Under the Guidance of
Prof. Sudipta Sen



**Department of Metallurgical and Materials Engineering
National Institute of Technology
Rourkela
2011**



**National Institute of Technology
Rourkela**

CERTIFICATE

This is to certify that the thesis entitled, “**Effect of Copper on the austempering behaviour of spheroidal graphite iron**” submitted by **Mr. KAUSIK TAMULI** and **Mr. AYAZ KHAN** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date:

Prof. Sudipta Sen

Dept. of Metallurgical and Materials Engineering
National Institute of Technology, Rourkela-769008

ACKNOWLEDGEMENT

With great pleasure, we would like to express our deep sense of gratitude to our guide **Prof. Sudipta Sen**, Department of Metallurgical & Materials Engineering, for his valuable guidance, constant encouragement and kind help at different stages for the execution of this dissertation work.

We are sincerely grateful to **Dr. B. B. Verma**, Head of the Department, Metallurgical & Materials Engineering, for providing valuable departmental facilities.

We would like to extend our sincere thanks to our project coordinators **Dr. Krishna Dutta** and **Dr. Archana Mallik** for helping us at each and every step in bringing out this report.

We would also like to thank **Mr. S. Hembrom** of Metallurgical and Materials Engineering Dept. for helping us out during different phases of my experimentation.

We are also very grateful to **Mr. Susanta Kumar Swain, Ph.D Scholar, Department of Metallurgical & Materials Engineering, National Institute of Technology, Rourkela** for helping us throughout the project and providing us with information and support as and when required.

NIT ROURKELA

Date:

KAUSIK TAMULI (107MM003)

AYAZ KHAN (1077MM05)

Bachelor of Technology

Dept. of Metallurgical and Materials Engineering

CONTENTS

TOPICS	Page No
ABSTRACT	iv
LIST OF FIGURES	v
CHAPTER 1: INTRODUCTION	1
CHAPTER 2: LITERATURE REVIEW	2
2.1 Spherodised Graphite Iron or Ductile Iron	2
2.1.1 <i>Composition of SG iron</i>	3
2.1.2 <i>Production of SG iron</i>	3
2.1.3 <i>Properties of SG iron</i>	4
2.1.4 <i>Effect of alloying elements on SGI</i>	4
2.2 Austempered Ductile Iron (ADI)	6
2.2.1 <i>Austempering</i>	6
2.2.2 <i>Effect of Cu</i>	8
2.2.3 <i>Microstructure</i>	8
2.2.4 <i>Production of ADI</i>	8
2.2.5 <i>Applications of ADI</i>	10
2.2.6 <i>Disadvantage of ADI</i>	10

CHAPTER 3: EXPERIMENTAL WORK	11
3.1 Specimen Preparation	11
3.1.1 <i>Specification of hardness testing specimen</i>	11
3.1.2 <i>Specification of tensile testing specimen</i>	11
3.2 Heat treatment / Austempering	12
3.2.1 <i>Austenitising</i>	12
3.2.2 <i>Quenching in a salt bath</i>	12
3.3 Metallography	13
3.4 Mechanical Testing	14
3.4.1 <i>Hardness test</i>	14
3.4.2 <i>Tensile test</i>	14
CHAPTER 4: RESULT AND DISSCUSSION	15
4.1 Hardness Test Results	15
4.1.1. <i>Discussion</i>	15
4.2 Tensile Test Results	16
4.2.1. <i>Discussion</i>	16
4.3 Graphs and Discussions	17
4.3.1 <i>Effect of austempering time on different mechanical properties</i>	17
4.3.1.1 <i>Effect of austempering time on hardness</i>	17
4.3.1.2 <i>Effect of austempering time on tensile strength</i>	18
4.3.1.3 <i>Effect of austempering time on yield strength</i>	19
4.3.1.4 <i>Effect of austempering time on elongation</i>	20
4.3.1.5 <i>Discussion</i>	21
4.3.2 <i>Effect of austempering temperature on different mechanical properties</i>	22
4.3.2.1 <i>Effect of austempering temperature on hardness</i>	22
4.3.2.2 <i>Effect of austempering temperature on tensile strength</i>	23
4.3.2.3 <i>Effect of austempering temperature on yield strength</i>	24

4.3.2.4 <i>Effect of austempering temperature on elongation</i>	25
4.3.2.5 <i>Discussion</i>	26
4.3.3 <i>Increase in UTS and YS of specimens with Cu over specimens without Cu</i>	27
4.3.3.1 <i>Increase in UTS with austempering time and temperature</i>	27
4.3.3.2 <i>Increase in YS with austempering time and temperature</i>	28
4.3.3.3 <i>Average increase in UTS and YS with austempering temperature</i>	29
4.3.3.4 <i>Discussion</i>	30
CHAPTER 5: CONCLUSION	31
CHAPTER 6: REFERENCES	32

ABSTRACT

Two types of spheroidal graphite cast iron samples, one with Cu and other without Cu were austempered at four different temperatures. The austempering temperatures were 250 °C, 300 °C, 350°C, 400°C. The main objective of the project was to develop the physical properties and morphology of the microstructures by austempering process. The tensile strength, yield strength, ductility, hardness and morphology of microstructure were studied after austempering. The effects of Cu on graphite nodule count, nodularity and pearlite percentage were observed. Samples with Cu showed higher values of tensile strength, yield strength, hardness but lower values of ductility as compared to samples without Cu. The effect of Cu on the formation of nodular graphite is not completely understood and much further work remains to be done. Both the types of irons have good nodular structure. Iron without Cu had a matrix of 95% ferrite and 5% pearlite. Iron with Cu % (0.42%) had about 50% ferrite and 50% pearlite as determined by using image analyzer. Thus offering all the production advantages of a conventional ductile iron casting, subsequently it is subjected to the austempering process to produce mechanical properties that are superior to conventional ductile iron.

LIST OF FIGURES

Page No

Fig 1. Diagram for austempering superimposed on TTT diagram	6
Fig 2. Schematic diagram for austempering treatment	9
Fig 3. Shape of tensile testing specimen.	11
Fig 4. Microstructure of as received specimen and ADI	13
Fig 5. Effect of austempering time on hardness at different austempering temperatures	17
Fig 6. Effect of austempering time on tensile strength at different austempering temperatures	18
Fig 7. Effect of austempering time on tensile strength at different austempering temperatures	19
Fig 8. Effect of austempering time on ductility at different austempering Temperatures	20
Fig 9. Effect of austempering temperature on hardness at different austempering times	22
Fig 10. Effect of austempering temperature on tensile strength at different austempering times	23
Fig 11. Effect of austempering temperature on yield strength at different austempering times	24
Fig 12. Effect of austempering temperature on ductility at different austempering times	25
Fig 13. Increase in tensile strength of specimen with Cu over in comparison to the specimen without Cu	27
Fig 14. Increase in yield strength of specimen with Cu in comparison to the specimen without Cu	28
Fig 15. Average increase in strength with temperature of specimens alloyed with Cu in comparison to the specimens without Cu	29

CHAPTER: 1

INTRODUCTION

Austempered ductile iron (ADI) is considered to be an important engineering material because of its attractive properties such as good ductility at high strength, good wear resistance and fatigue strength and fracture toughness^[1]. Because of these combinations of properties, ADI is now used extensively in many structural applications in automotive industry, defense and earth moving machineries.^[1] The optimum mechanical properties of ADI i.e., the adequate combination of strength, toughness, fatigue strength, and wear resistance could be achieved if the microstructure consists of retained carbon-enriched stable austenite (enables ductility), together with one of two bainitic morphologies, namely, carbide-free bainitic ferrite or bainitic ferrite, in which carbides are distributed in the ferrite (affects strength)^[2].

The mechanical properties of ADI depend on the microstructure, which in turn depends on the austempering variables, i.e. austempering temperature and the time of holding^[4]. In conventional ductile iron the mechanical properties can be attributed to the pearlite and the ferrite present in the matrix but the superiority in the mechanical properties of the ADI are due to the acicular ferrite and carbon enriched stabilized austenite present in the matrix^[2]. The proportion in which these two phases are present depends on the austempering variables.

The base iron chemistry and the alloy additions in ductile iron, plays important role in ADI technology. Most of the ADI needs to be alloyed for satisfactory austemperability and subsequent improvement in properties^[5]. Now-a-days many researches for ADI are done to study the effect of the alloying elements on the microstructure, mechanical properties. As an alloying element, copper widens the austenite zone of the phase diagram increasing the transformation rate during austenitising process and the carbon content in the matrix. On the other side, during the austempering process, copper may subdue carbide formation^[4].

In the present work, the effect of the austempering variables, i.e. time of holding and temperature of austempering, on the mechanical properties are studied for ADI when alloyed with Cu.

CHAPTER: 2

LITERATURE REVIEW

2.1 Spherodised Graphite Iron or Ductile Iron:

Ductile iron, also referred to as nodular or spheroidal graphite cast iron constitutes a family of cast irons in which the graphite is present in a nodular or spheroidal form. Ductile iron derives its name from the fact that, in the as-cast form, it exhibits measurable ductility. By contrast, neither white cast iron nor grey cast iron exhibits significant ductility in a standard tensile specimen^[9]. It is produced by special alloy addition and proper cooling rates so that the carbon can be converted to spherical forms. The nodules are formed during solidification and not during heat treatment. The graphite nodules are small and constitute only small areas of weakness in a steel matrix. Because of this, the mechanical properties of ductile irons are related directly to the strength and ductility of the matrix present. The matrix of ductile irons can be varied from a soft and ductile ferritic structure, through harder and higher strength pearlitic structures to a hard, higher and comparatively tough tempered martensitic or bainitic structure. Thus, a wide range of combinations of strength and ductility can be achieved by alloying and heat-treating the ductile iron.

Depending upon the matrix phases present, SG iron can be classified into four groups namely; ferritic, pearlitic, martensitic and austenitic. Generally, SG irons are ferritic but due to its low yield strength and high ductility, its use is limited for certain applications. Thus, some carbon is left intentionally to form some cementite and get enhanced properties. They are referred as pearlitic SG iron. If the cooling rate is higher than the critical cooling rate, then the matrix will be martensitic. Thus, the matrix may vary from a soft ductile ferritic structure through a hard and higher strength pearlitic structure to an austenitic structure.

2.1.1 Composition of SG iron:

Carbon contents of unalloyed ductile iron ranges from 3.0 wt. % to 4.0 wt. % and silicon content from 1.6 wt. % to 2.8 wt. %. Manganese can vary from 0.1 wt.% to 1.0 wt.%, phosphorous from 0.01 wt.% to 1.0 wt.% and sulphur should be maximum 0.03 wt.%^[8,9]. All elements in the composition of ductile iron should be controlled.

2.1.2 Production of SG iron:

1. *Desulphurization*: Since sulphur helps in the growth of graphite flakes, hence SG iron should have low sulphur content (<0.1%). Sulphur can be removed during melting or by addition of a desulphurising agent like calcium carbide or soda ash.

2. *Nodulising*: Magnesium reacts with oxygen to form highly stable MgO which floats on the surface and can be skimmed off easily. Oxygen content thus reduces from typical levels of 90-135ppm to about 15-35ppm. Magnesium also reacts with sulphur to produce less stable MgS. As Mg is less soluble in the metal and it is volatile, hence reverse reaction can take place if losses are not taken care of. Si is added for further deoxidation. Cerium forms highly stable oxides with S and O and is less volatile than Mg. Addition of Mg is done when the melt is at 1500°C but Mg vaporizes at 1100°C. Magnesium being lighter floats on the bath and being reactive burn off at the surface. So Mg is added as Ni-Mg, Ni-Si-Mg alloy or magnesium coke to reduce the violence of the chemical reaction and to have saving in Mg. After nodulising treatment inoculants like Mg have their spherodising effect on the graphite structure so that graphite nodules can be formed.

3. *Inoculation*: Inoculation increases the number of points available for the precipitation and subsequent growth of graphite. It involves the addition of small amounts of a material (inoculants) to the molten metal either just before or during pouring. It is seen that high levels promote graphite structure whilst low levels can result in the formation of either mottled structure or white irons. The need for a high level of nucleation increases as cooling rate increases. In addition to its effect on graphite morphology, Mg is a powerful carbide promoter and as a result, compared with the gray irons, there is a far greater tendency for ductile irons to solidify with white or mottled structure. The primary purpose of inoculating ductile irons is to suppress formation of chill and mottle. In addition, inoculation is important in maintaining good nodule shape and also high nodule numbers. All effective inoculants are based on silicon. The most widely used is foundry grade ferrosilicon, containing about 75% silicon. The inoculating effect produced initially increases as the amount of inoculants is increased, but the effect soon begins to

level off. A situation is reached where the extra inoculating benefit obtained is too small to justify for the increased addition.

4. *Solidification*: Solidification of SG iron is always associated with under cooling. Graphite nuclei grow slowly and then are surrounded by austenite. The combination of austenite and graphite corresponds to the eutectic point at eutectic temperature. Austenite which gets supersaturated with carbon cools and a new equilibrium is established at the graphite-austenite interface. The excess of carbon diffuses towards the graphite nodule where it precipitates out.

2.1.3 Properties of SG iron:

The properties of ductile iron can vary depending on its grade. The tensile strength can be as low as 400 MPa for ferritic grades and can go as high as 1300 MPa for austempered ductile iron grades. The yield strength can also vary over a large range, between 250 MPa to 800 MPa. The elongation can sometimes be as high as 25%, but it is possible for ferritic grades only. High fluidity of ductile iron enables it to be casted easily. It has very good machinability because of the graphite present which makes chip formation easier. Ductile iron is highly corrosion resistant^[10,11].

2.1.4 Effect of alloying elements on SGI:

1. *Silicon*: Addition of Silicon results in the presence of both ferrite and pearlite in the matrix. Silicon enhances the performance of ductile iron at elevated temperature by stabilizing the ferritic matrix and forming a silicon rich surface layer, which inhibits oxidation.

The detrimental effects of increasing silicon content are:

- i) Reduction in impact energy.
- ii) Increase in impact transition temperature.
- iii) Decrease in thermal conductivity.

Si is used to promote ferrite and to strengthen ferrite. So Si is generally kept below 2.2% while producing ferritic grades and between 2.5% and 2.8% when producing pearlitic grades.

2. *Manganese*: Mn is a mild pearlite promoter. Improves properties like proof stress and hardness to a small extent. Mn retards the onset of the eutectoid transformation, decreases the rate of diffusion of carbon in ferrite and stabilizes cementite (Fe_3C). Its presence can sometimes result in embrittlement.

3. *Copper*: Cu is a strong pearlite promoter. It increases the proof stress as well as the tensile strength and hardness with no embrittlement in the matrix. So in the pearlitic grade of the ductile iron, 0.4-0.8% of Cu is added.

4. *Nickel*: Ni helps in increasing the U.T.S without affecting the impact values. It strengthens ferrite, and reduces ductility, but to a much lesser extent than that of silicon. It is added in the range of 0.5-2.0%. For additions in excess of 2%, there is chance of embrittlement.

5. *Molybdenum*: Mo is a mild pearlite promoter. Mo increases proof stress and hardness. It forms intercellular carbides and there is a danger of embrittlement and can sometimes result in low tensile strength and ductility. Mo also improves elevated temperature properties.

6. *Chromium*: Cr prevents corrosion by forming a thin layer of chromium oxide over the surface and stops further exposition of the surface to the atmosphere. But as it is a strong carbide former, hence it is not desirable in carbide free structures.

7. *Magnesium*: Magnesium works as the modifier in the matrix and nodularizes the graphite so as to increase the ductility and yield strength.

8. *Sulphur and Phosphorus*: Phosphorous is intentionally kept very low because its presence causes cold shortness and the property of ductile iron gets impaired. Addition of Sulphur is done for better machinability but is kept low, a maximum of 0.015%, as larger sulphur addition may cause the hot (red) shortness.

Austempered Ductile Iron is a subclass of spherodised graphite iron which has superior properties. The heat treatment process of austempering is necessary to get this type of cast iron. It is discussed in details in next section.

2.2 Austempered Ductile Iron(ADI):

Austempered Ductile Iron is a ductile iron that has undergone an isothermal heat treatment called austempering.

2.2.1 Austempering:

Austempering is the heat treatment process in which austenite transforms isothermally to lower bainite and thus is used objectively to reduce distortion and cracks^[12]. In austempering the steel is heated to the austenitic range and then the steel is quenched in molten salt bath held at a temperature above M_s and the austenite at this temperature is let to transform to lower bainite.

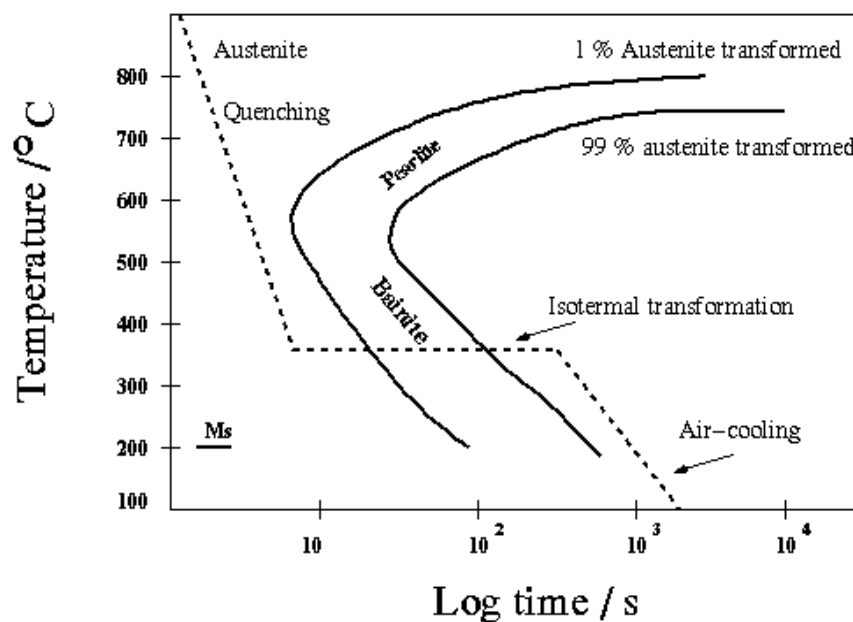


Fig 1. Diagram for austempering superimposed on TTT diagram

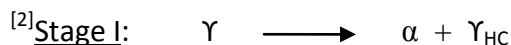
The steel to be austempered should have adequate hardenability so as to avoid pearlitic transformation when the steel is quenched from the austenitic range into a heated molten bath maintained at a temperature above M_s . Moreover the bainitic bay should not be long, else bainitic transformation will be incomplete. The major processing advantage of austempering is that it does not require tempering^[12].

Austempering results in enhanced ductility, enhanced toughness, higher hardness and lesser distortion and quench cracks than that observed after tempering of hardened specimen^[12]. Combination

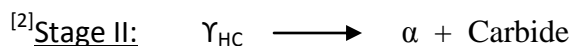
of attractive properties such as good ductility at high strength, good wear resistance and fatigue strength and fracture toughness can be obtained for ADI^[1]. The desired mechanical properties of ADI i.e., the adequate combination of strength, toughness, fatigue strength and wear resistance could be achieved by varying the austempering variables, i.e. the temperature at which austempering is done and the time of holding.

The attractive properties of ADI are due to the uniqueness in its microstructure which consists of ferrite (α) and high carbon austenite (γ_{HC}). This is different from the austempered steels where the microstructure consists of ferrite and carbide. The product of austempering reaction in ductile iron is often referred to as ausferrite rather than bainite^[1]. The high silicon content of the ductile iron suppresses the precipitation of carbide during austempering reaction and retains substantial amount of stable high carbon austenite (γ_{HC})^[1,2]. During austempering, the bainitic ferrite forms by rejection of carbon into the residual austenite. As austempering progresses, more of bainitic transformation occurs accompanied by rejection of more carbon into the surrounding austenite resulting in the increase in the amount of austenite and the amount of carbon in the austenite. In earlier stages, the carbon content of austenite is insufficient to make it stable, and therefore, it transforms to martensite. However, at longer times austenite is enriched to the extent that it can become thermally stable to well below room temperature^[5].

The bainitic transformation in the austempered ductile iron can be described as a two stage phase transformation reaction. The initial transformation is of primary austenite (γ) decomposing to ferrite (α) and high carbon-enriched stable austenite (γ_{HC}). This transformation is commonly known as the stage I reaction^[2].



If the casting is held at the austempering temperature for too long, then a second reaction (stage II) sets in, where high-carbon austenite further decomposes into ferrite and carbide^[2].



Stage II reaction is undesirable since it causes the embrittlement of structure and degrades the mechanical properties of ADI^[2]. The carbide formed is ϵ carbide which makes the steel brittle^{[1],[11]}. Since, ϵ carbide is a detrimental phase constituent, hence this reaction during austempering process must be prevented.

The best combination of mechanical properties in ADI can be obtained after the completion of the first stage reaction but before the onset of the second reaction. The time interval between the completion of the first reaction and the onset of the second reaction is termed as “process window”. In order to enlarge this process window alloying elements are added^[1,14,15].

2.2.2 Effect of Cu:

Alloying elements are added in order to delay the transformation of austenite in ductile iron. Depending upon the relative effectiveness of the alloying elements on the reactions in stage I and stage II of the austempering process, they are added^[5]. Cu does not alter the carbon diffusion in austenite or the stability of austenite. It has been reported that Cu suppresses carbide formation in lower bainite^[5].

Copper widens the austenite zone of the phase diagram, increasing both the transformation rate during austenitising process and the carbon content in the matrix. Again, during the subsequent austempering process, copper may restrain carbide formation^[6]. Hence, stage II reaction may be delayed due to Cu addition and result in prevention from deterioration of properties.

2.2.3 Microstructure:

During austempering, as the phase transformation progresses, austenite transforms to bainitic ferrite by rejection of carbon to the austenitic region. For short austempering times, the formation of martensite cannot be prevented during the subsequent cooling from the austempered temperature to room temperature. This is because the austenite is not sufficiently enriched with carbon to stabilize it. With longer austempering times, the martensite disappears from the structure, whereas the amount of bainitic ferrite and retained austenite increases. But at still higher austempering times, the amount of retained austenite decreases and this leads to lower ductility and impact energy values^[6].

2.2.4 Production of ADI:

Austempered ductile iron castings must be free from surface defects, carbides, porosity, inclusions and should have a consistent chemical composition. The composition of ADI casting is little different from that of conventional ductile iron casting. While selecting the composition for ADI, care should be taken that the elements which are detrimental to the casting quality are controlled. It should be seen that the iron is sufficiently alloyed to avoid pearlitic transformation but not over alloyed, else

bainitic bay may be too long and long holding time at the bath temperature will be required. The microstructure should be free from intercellular carbides.

A typical composition of ductile iron casting used for making ADI is: carbon in the range of 3.5% to 3.7%, Si in the range of 2.5% to 2.7%, Mn in the range of 0.25% to 0.31%, Cu between 0.05% to 0.8%, Ni between 0.01% to 0.8% and a maximum of 0.25% of Mo if required^[16].

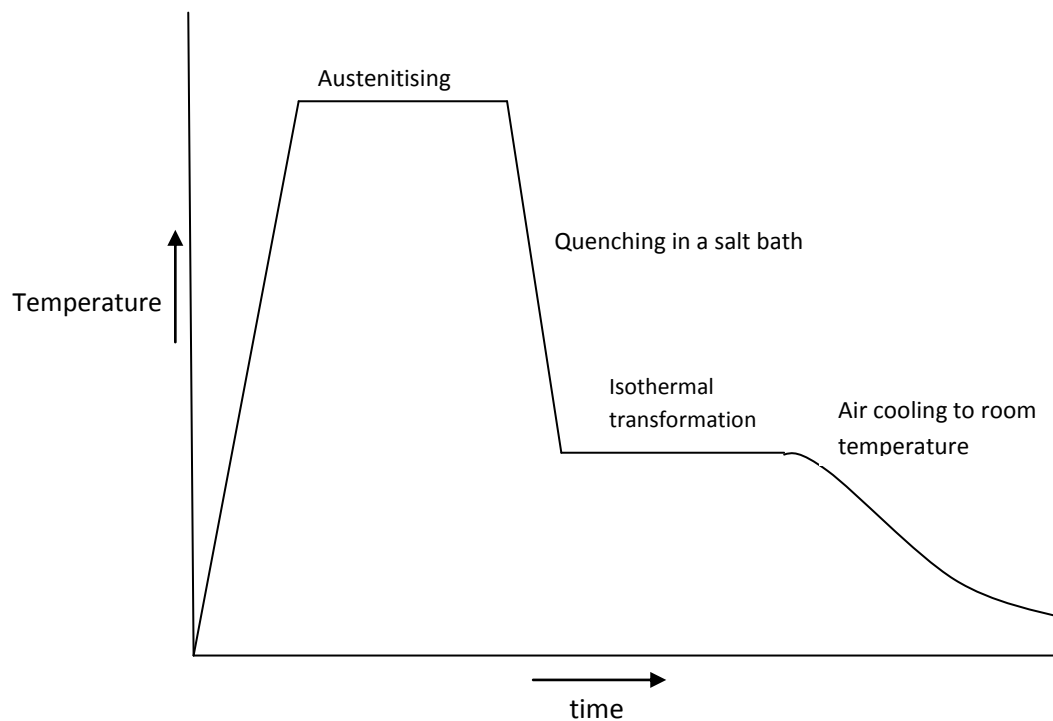


Fig 2. Schematic diagram for austempering treatment

Austempered ductile iron is produced by the isothermal heat treatment process called austempering, of ductile iron. It under goes the following steps:

1. *Austenitising*: The ductile cast iron is heated to the austenitising temperature in the range of 850°C to 950°C. The heating is done in a furnace for about 1 to 2 hours so that the entire part of the casting gets converted to austenite.
2. *Austempering*: The austenitised part is rapidly quenched in a salt bath to avoid pearlitic transformation. Quenching is done from the austenitising temperature of 850°C - 950°C in a salt bath maintained at a temperature of 200°C - 450°C.
3. *Holding time*: The casting is hold at the desired temperature for sufficient time to allow bainitic transformation to be complete. The time of holding depends on the austempering temperature and can vary from 0.5 hour to 3 hours.
4. *Air cooling*: After holding the sample for sufficient time so that the bainitic transformation has taken place, it is air cooled to the room temperature.

2.2.5 Applications of ADI:

Austempered ductile iron is used in gears in automobiles; crankshafts for high powered diesel engines, air conditioning and refrigerator applications; suspensions; axel boxes of railway engine and pick up arms for railway track; agricultural applications; various hardware for trucks and armored vehicles in defense industry.

2.2.6 Disadvantage of ADI:

A major disadvantage of ADI is that welding is not recommended for it. Another disadvantage is that higher hardness grades must be machined before heat treatment although the low hardness grades can be machined after heat treatment^[17].

CHAPTER: 3

EXPERIMENTAL WORK

3.1 Specimen Preparation :

Spherodised Graphite iron specimens were collected. Specimens for carrying out the hardness test as well as tensile testing specimen were collected.

3.1.1 Specification of hardness testing specimen:

Specimens of two type of composition were collected. 1st type, not having Cu as one of its constituent elements and the other type, having Cu in the range of 0.3% to 0.6%. 15 specimens of each of the type were collected. The specifications of all the 30 specimens were roughly the same, i.e. 10 mm x 10 mm x 25 mm.

3.1.2 Specification of tensile testing specimen:

The tensile testing specimen as per the Indian Standard is collected. 12 specimens having Cu in the range of 0.3% to 0.6% as one of its constituent elements and 12 other specimen which do not have Cu as one of its constituent elements are collected.

The specification is as per the ISO 1608: 1995 ANNEX C.

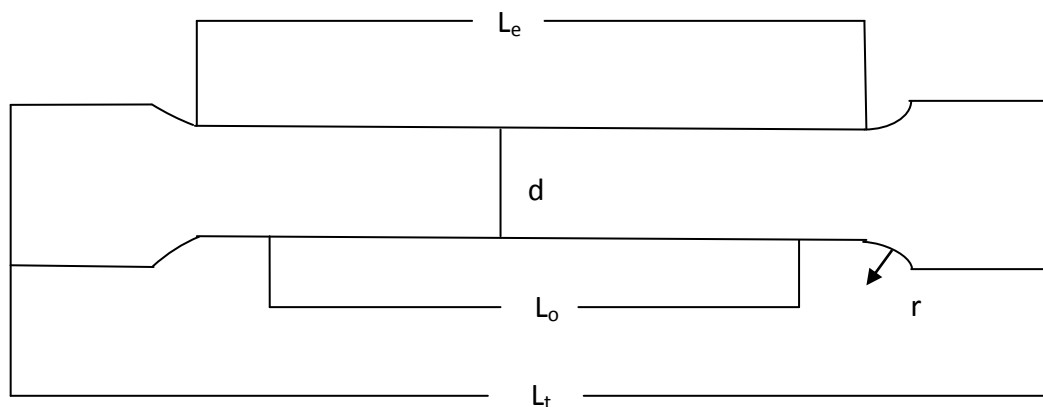


Fig 3. Shape of tensile testing specimen.

L_o = original gauge length

d = diameter of the parallel section

As per ISO 1608:1995 ANNEX C,

$L_o = k \sqrt{S_o}$ where S_o is the original cross-sectional area and k is a constant equal to 5.65

Hence, for $d = 10$ mm, $L_o = 50$ mm.

L_e = parallel length of the machined test piece = $L_o + d/2 = 55$ mm.

L_t = total length of the machined test piece = $L_o + 2d = 70$ mm.

for $d = 10$ mm, $r = 8$ mm (As per ISO 1608:1995. Table 5)

3.2 Heat treatment / Austempering:

3.2.1 Austenitising:

The test specimens were austenitised in a muffle furnace.

Austenitising temperature = 850°C

Time of holding = 1 hour

3.2.2 Quenching in a salt bath:

The austenitised specimens were quenched in a salt bath maintained at 4 different temperatures. 3 specimens at a time were austenitised and after austenitising them, they were immediately transferred from the muffle furnace to the salt bath. After a time interval of 30 minutes 1 of the specimen was taken out from the salt bath and air cooled. After next 30 minutes the other specimen was air cooled and after 90 minutes the last specimen was air cooled. This process is carried out for both hardness testing and tensile testing specimens and for both specimens with Cu and without Cu.

Salt bath composition = 50 % NaNO_3 + 50 % KNO_3

Austempering temperatures = 250°C, 300°C, 350°C and 400°C

Holding time = 0.5 hour, 1 hour and 1.5 hour

3.3 Metallography:

3 cuboidal specimens were used for metallography. 1 in the as received condition, 1 without Cu austempered at 250°C for 0.5 hour and 1 with Cu austempered at 250°C for 1.5 hour were polished and etched and microstructure viewed in optical microscope.

Etchant used = 3% Nital (3% HNO_3 + 97% $\text{C}_2\text{H}_5\text{OH}$)

The SEM pictures of the specimens with and without Cu are taken.

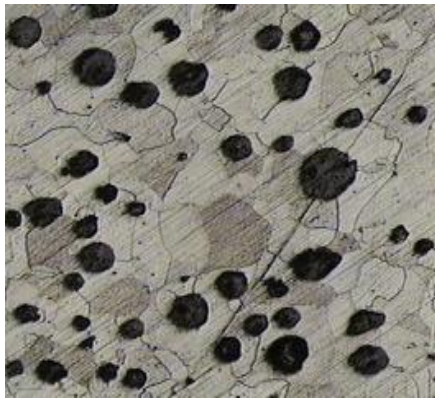


Fig 4 (a)

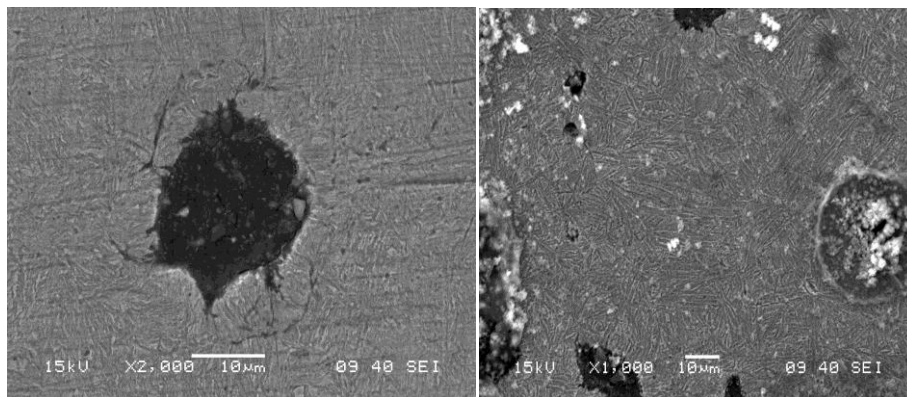


Fig 4 (b)

Fig 4 (c)

Fig 4. Microstructures of a) as received specimen b) ADI without Cu c) ADI with Cu

3.4 Mechanical Testing:

3.4.1 Hardness test:

The hardness of the as received specimen as well as that of all the austempered specimen was measured using Rockwell Hardness tester. Hardness was measured in Rockwell A scale. The Rockwell A scale utilizes a diamond penetrator and a major load of 60 kg.

The hardness testing specimen was rubbed with an emery paper. The specimen was seated on the specimen holder. The face of the specimen which remains in contact with the specimen holder should be parallel to the surface of the holder. Also the face on which indentation is to be made should be plane. Now, a minor load of 10 kg is applied by rotating the axel. The axel should be rotated untill the reading on the display is zero. After that, a major load of 60 kg is applied on the specimen by pressing the loading button. Reading on the scale A was taken as the hardness value. For each specimen, hardness value was found out on two to three different places and the average value was taken as the hardness of the specimen.

3.4.2 Tensile test:

The instrument used for tensile testing is Universal Testing Machine INSTRON 1195.

Using a electronic slide caliper the thickness and the total length of the specimen was measured. The diameter of the specimen and the gauge length which was fixed at 50mm was fed to the testing machine. The distance between the jaws was fixed according to the gauge length of the specimen. The specimen was gripped by the jaws and axial load was applied to it.

Full scale load was fixed at 100 kN.

Cross-head speed = 5 mm/min

Loading was done till the specimen fails. The readings corresponding to yield strength, tensile strength for each specimen was noted.

The failed parts of the specimen are taken and joined together and the length of the specimen was measured. The elongation of the specimen was measured and the % elongation calculated.

CHAPTER: 4

RESULTS AND DISCUSSION

4.1 Hardness Test Results:

TABLE 1.

Serial no	Austenitising temperature=850°C; Austenitising time = 1 hour		Hardness Value	
	Austempering Temperature	Austempering Time	Without Cu	With Cu
1	250 °C	0.5 hour	R _A 56	R _A 63
2		1 hour	R _A 64	R _A 73
3		1.5 hour	R _A 62	R _A 71
4	300 °C	0.5 hour	R _A 54	R _A 59
5		1 hour	R _A 63	R _A 69
6		1.5 hour	R _A 61	R _A 68
7	350 °C	0.5 hour	R _A 53	R _A 56
8		1 hour	R _A 61	R _A 67
9		1.5 hour	R _A 59	R _A 65
10	400 °C	0.5 hour	R _A 46	R _A 54
11		1 hour	R _A 56	R _A 63
12		1.5 hour	R _A 53	R _A 60
	As received condition		R _A 42	R _A 43

4.1.1. Discussion:

The hardness value of the specimens with Cu gets increased as compared to the specimen without Cu in ADI by 3 to 9 Rockwell Hardness unit in A scale for different austempering conditions. In as received condition, Cu hardly influences the hardness. This increase in hardness over the specimen without Cu is due to the presence of large amount of pearlite in the matrix of the specimen alloyed with Cu.

4.2 Tensile Test Results:

TABLE 2.

Serial no	Austenitising temperature=850°C; Austenitising time = 1 hour		UTS (in Mpa)		Yield Strength (in Mpa)		Elongation (%)	
	Austempering Temperature	Austempering Time	Without Cu	With Cu	Without Cu	With Cu	Without Cu	With Cu
1	250 °C	0.5 hour	927	965	689	719	2.1	1.7
2		1 hour	1077	1122	847	884	2.6	2.3
3		1.5 hour	1065	1105	821	851	2.8	2.4
4	300 °C	0.5 hour	767	815	529	579	3.7	3.4
5		1 hour	921	973	686	732	4.3	4.1
6		1.5 hour	901	957	648	684	4.6	4.3
7	350 °C	0.5 hour	649	711	434	502	6.1	5.7
8		1 hour	809	869	561	634	6.9	6.4
9		1.5 hour	795	855	539	608	7.1	6.5
10	400 °C	0.5 hour	588	656	379	451	5.5	5.2
11		1 hour	766	815	521	592	5.8	5.6
12		1.5 hour	742	792	497	576	5.6	5.5
	As received condition		556	593	324	383	23.2	8.4

4.2.1. Discussion:

It is seen that tensile strength and yield strength of the specimens with Cu gets increased as compared to the specimens without Cu but Cu decreases ductility of ADI by some nominal value. This is because, Cu is a pearlite promoter and the matrix of the ADI with Cu as the alloying element contains much large amount of pearlite but that without Cu contains large amount of softer phase, ferrite.

4.3 Graphs and Discussions:

4.3.1 Effect of austempering time on different mechanical properties:

4.3.1.1 Effect of austempering time on hardness:

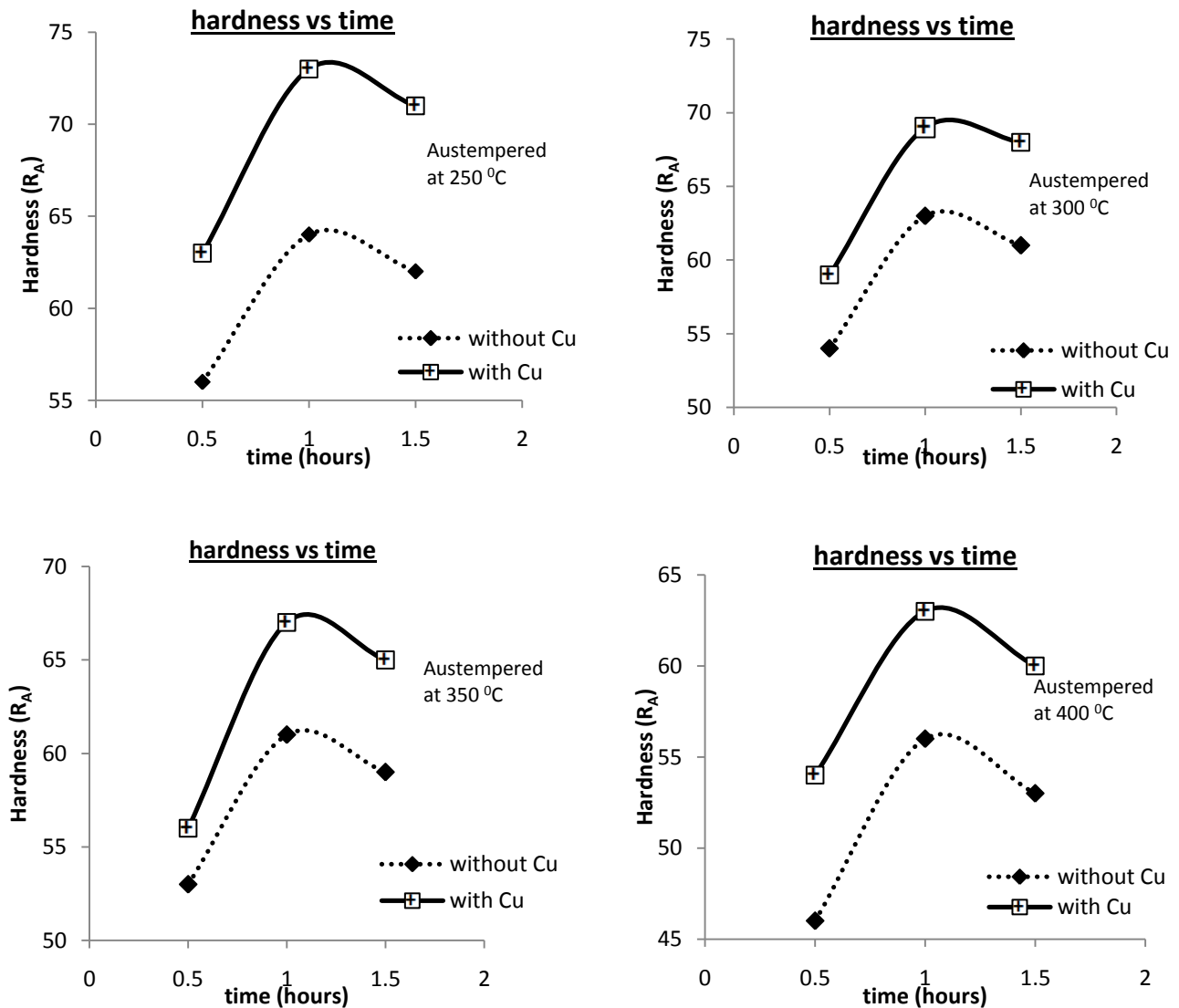


Fig 5. Effect of austempering time on hardness at different austempering temperatures

4.3.1.2 Effect of austempering time on tensile strength:

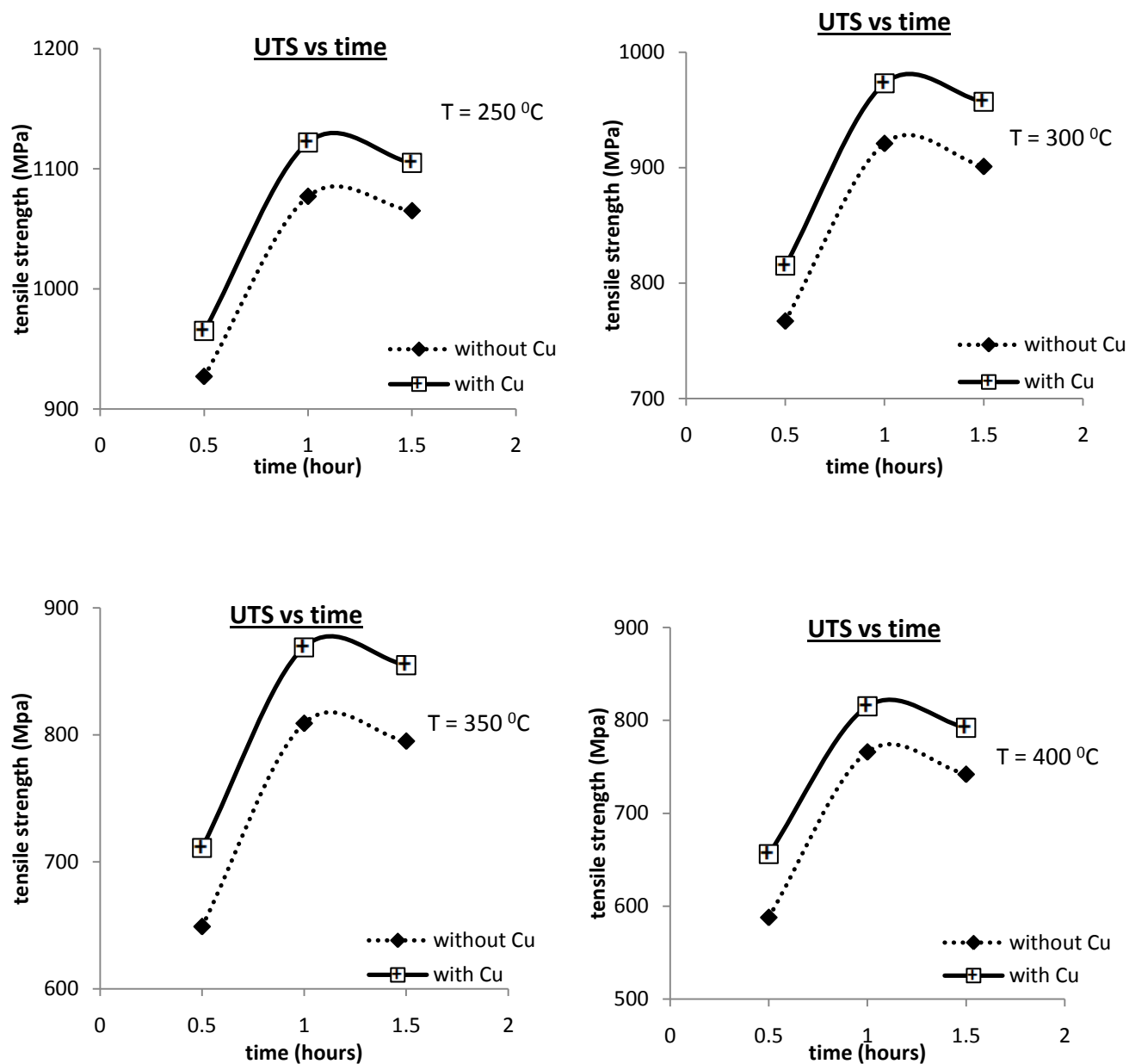


Fig 6. Effect of austempering time on tensile strength at different austempering temperatures

4.3.1.3 Effect of austempering time on yield strength:

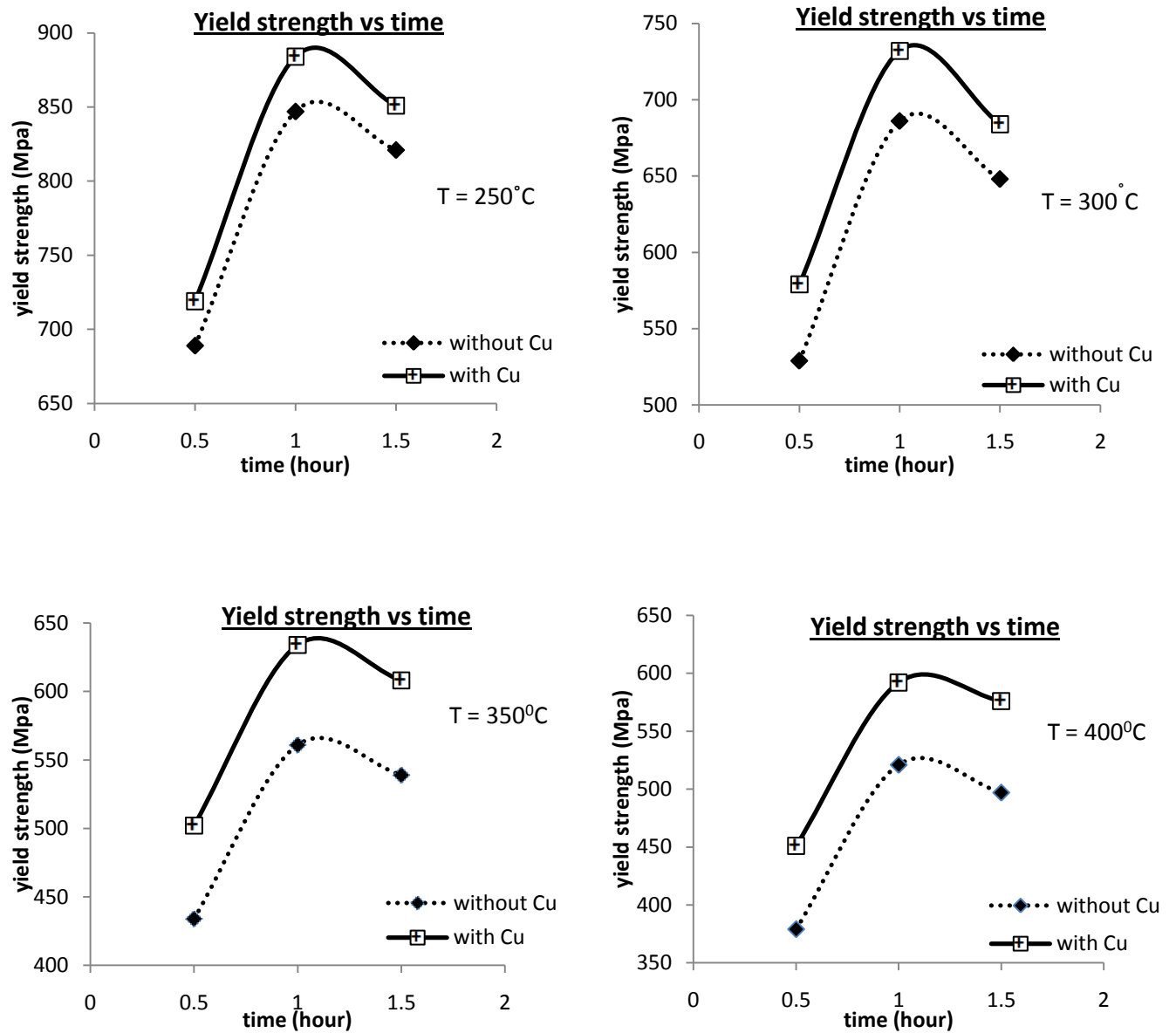


Fig 7. Effect of austempering time on tensile strength at different austempering temperatures

4.3.1.4 Effect of austempering time on elongation:

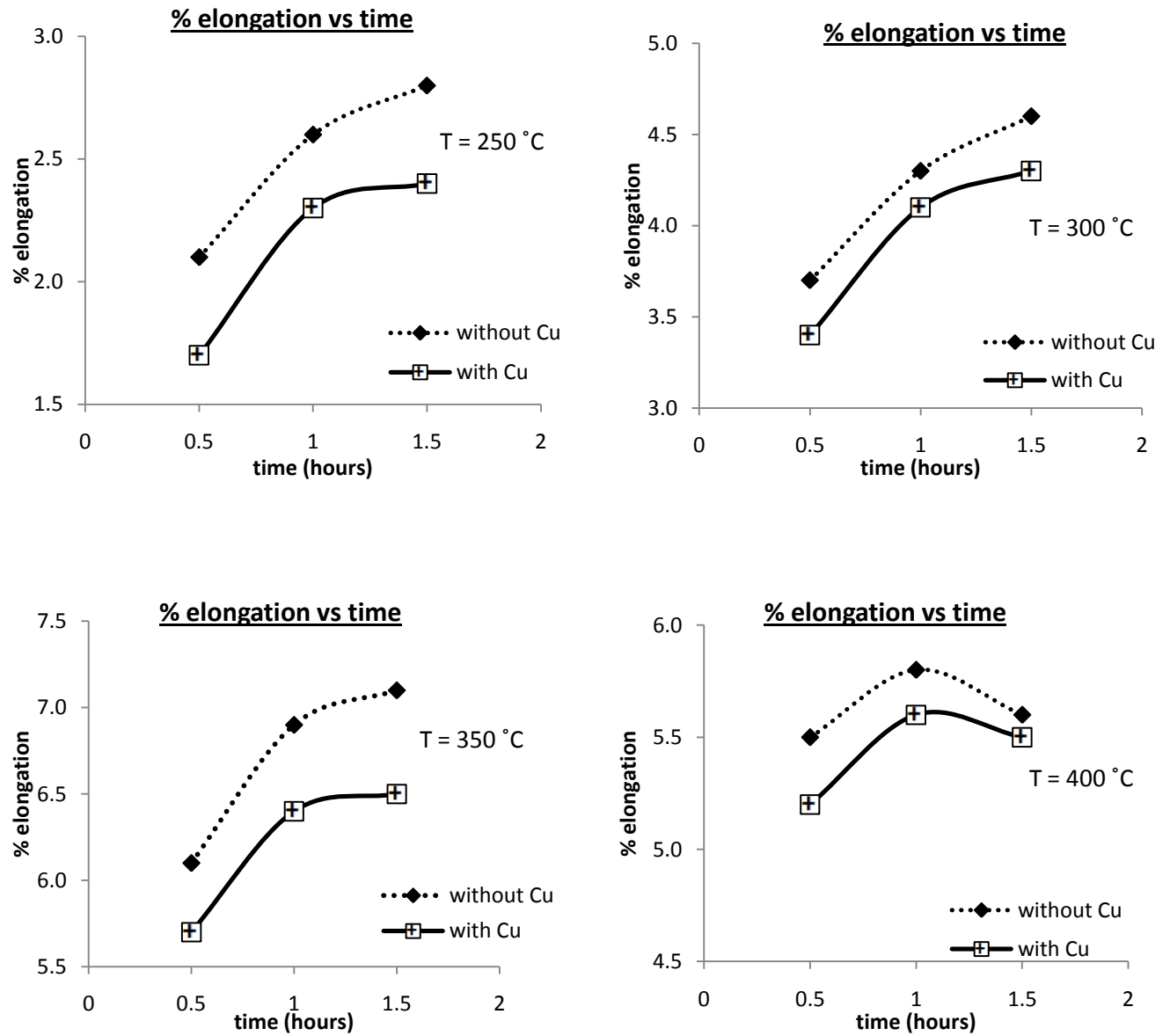


Fig 8. Effect of austempering time on ductility at different austempering temperatures

4.3.1.5 Discussion:

The hardness, tensile strength and yield strength of the specimen with Cu gets increased as compared to the specimens without Cu but the Cu decreases ductility in ADI by some nominal value.

For smaller austempering times, during the initial stage, stage I reaction proceeds and the amount of bainitic ferrite and high carbon austenite gradually increases. But carbon enrichment in retained austenite is too less to make all the retained austenite stable at room temperature and some transformation to martensite is involved. With the increase in austempering time, the amount of retained austenite and bainitic ferrite increases until completion of bainitic transformation resulting in increase in hardness, tensile strength and yield strength. After completion of bainitic transformation, if austempering is continued for still longer duration, stage II reaction sets in and retained austenite decomposes to bainitic ferrite and carbide. This results in decrease of hardness, tensile strength and yield strength after achieving a peak value.

The low ductility for shorter austempering times can be attributed to some brittle fracture taking place due to the presence of martensite in the microstructure. But with the increasing austempering time, the amount of retained austenite increases, resulting in increase of elongation. This reaches maximum at the completion of stage I reaction and with the onset of stage II reaction the ductility decreases owing to the decrease in retained austenite.

4.3.2 Effect of austempering temperature on different mechanical properties:

4.3.2.1 Effect of austempering temperature on hardness:

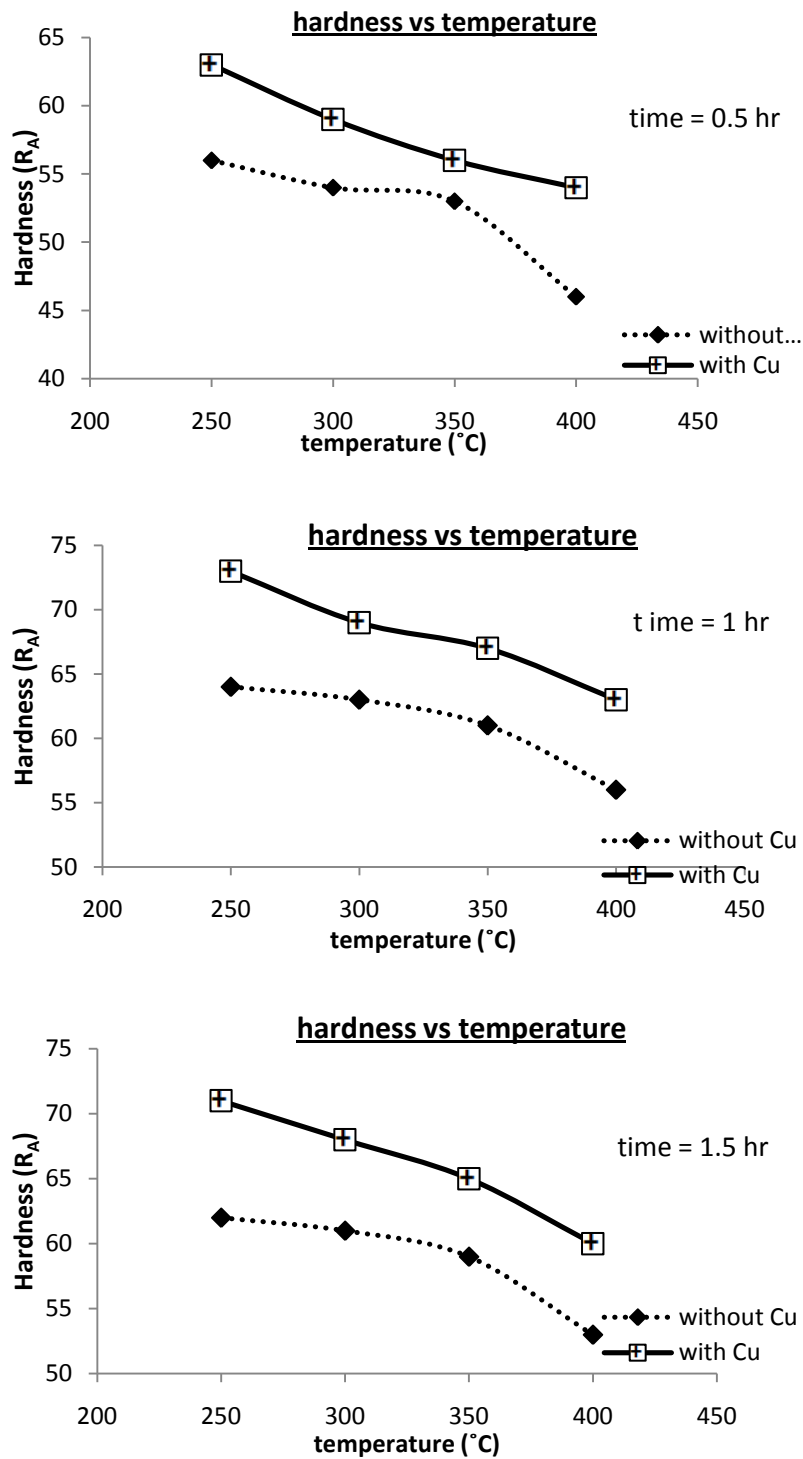


Fig 9. Effect of austempering temperature on hardness at different austempering times

4.3.2.2 Effect of austempering temperature on tensile strength:

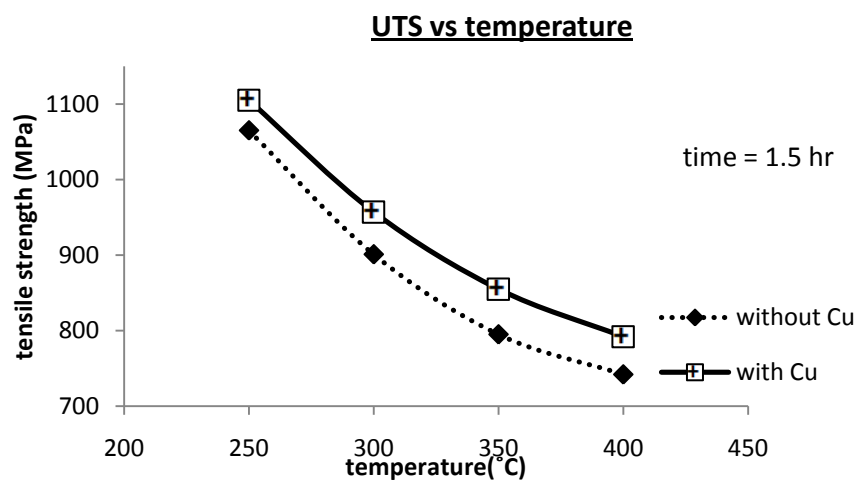
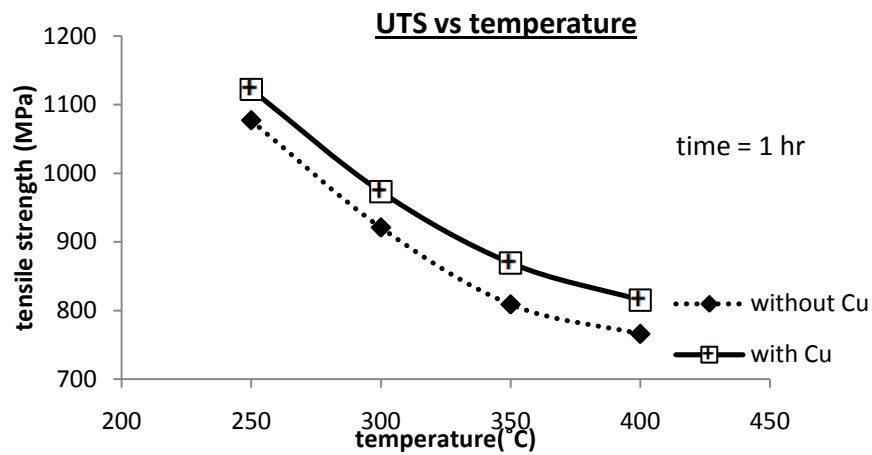
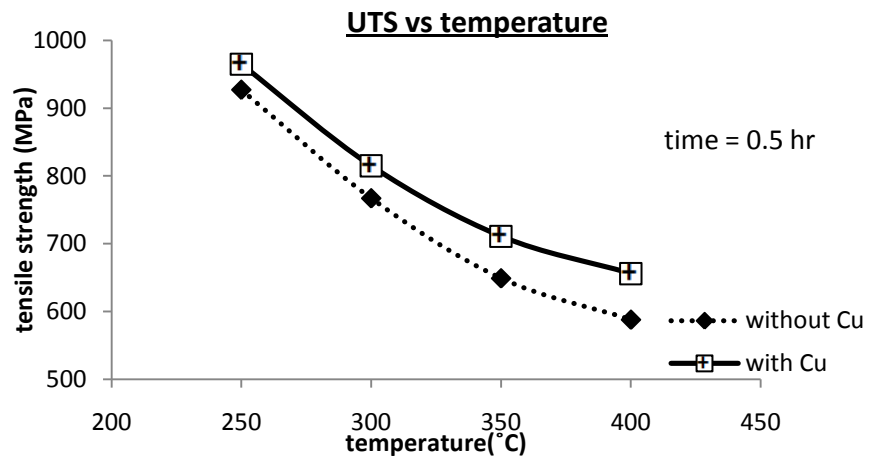


Fig 10. Effect of austempering temperature on tensile strength at different austempering times

4.3.2.3 Effect of austempering temperature on yield strength:

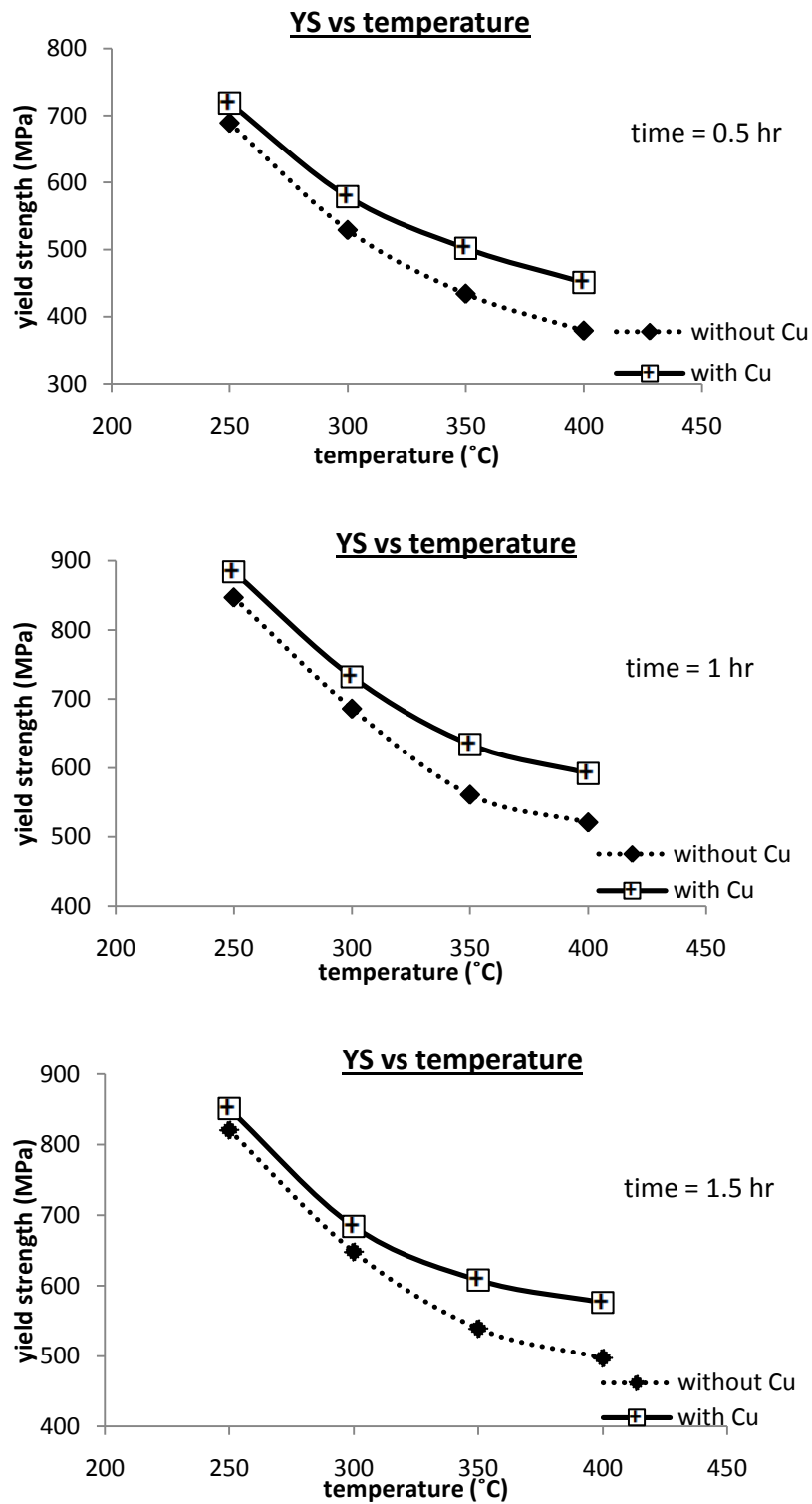


Fig 11. Effect of austempering temperature on yield strength at different austempering times

4.3.2.4 Effect of austempering temperature on elongation:

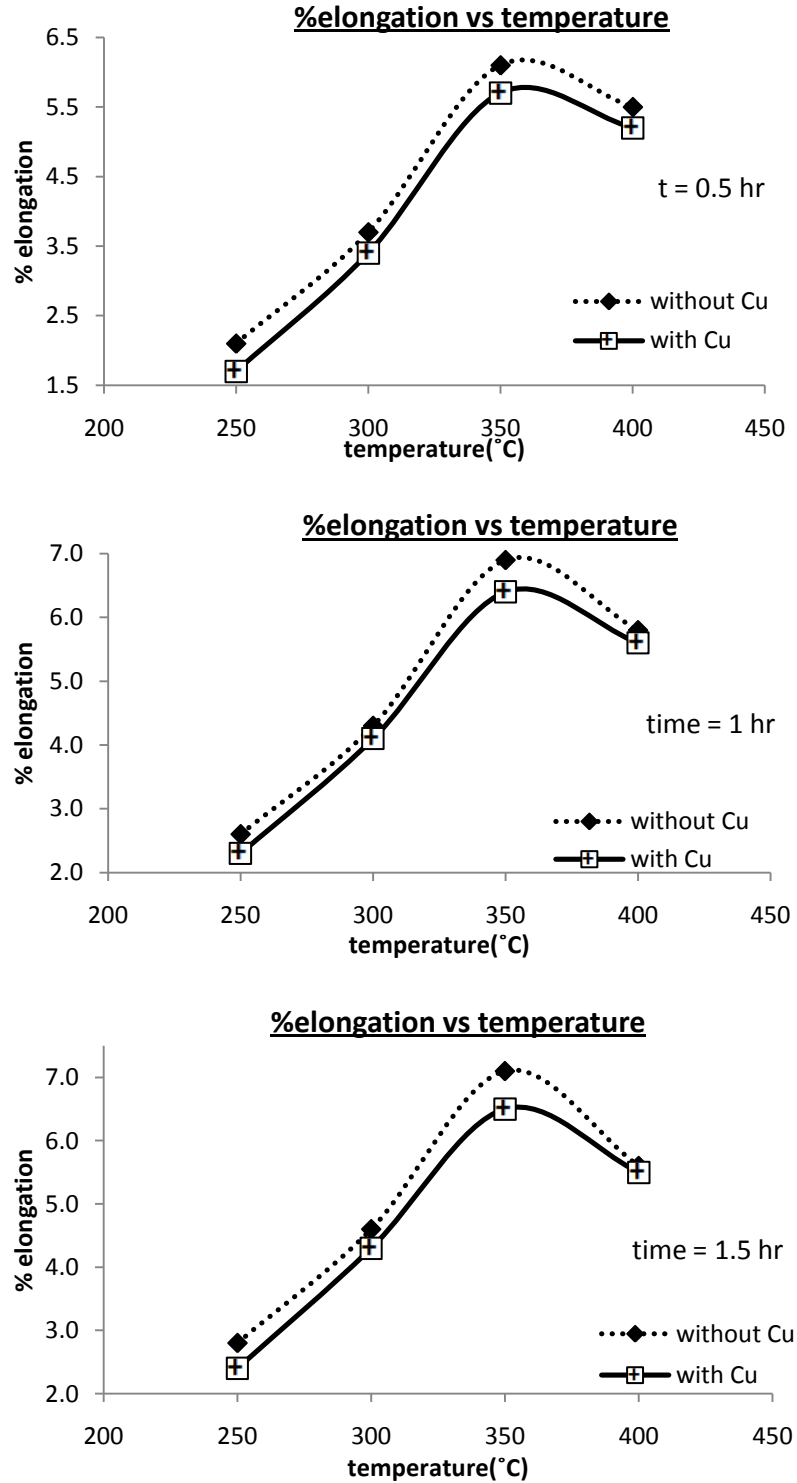


Fig 12. Effect of austempering temperature on ductility at different austempering times

4.3.2.5 Discussion:

The hardness, tensile strength and yield strength of the austempered ductile specimen decreases with the increase in temperature but the ductility initially increases with temperature and then after reaching a peak value it starts decreasing.

At lower austempering times, the high strength and high hardness value can be attributed to the presence of acicular bainite and some martensite and retained austenite. The fine structure of the bainite plates and low amount of retained austenite results in high strength at low austempering temperature. Moreover, other factors such as dispersed carbides, high dislocation density and lattice distortion of the ferrite contribute to the mechanical properties. With the increase in austempering temperature, the amount of retained austenite increases and martensite disappears from the microstructure resulting in decrease in strength and hardness. At higher austempering temperature, bainitic ferrite produced is coarser but in lesser volume, leading to decrease in strength.

The low ductility of the austempered ductile iron at low austempering temperature can be attributed to the brittle fracture occurring due to the presence of martensite in the microstructure. Moreover, the amount of retained austenite at low temperature is less resulting in lesser elongation. With the increase in austempering temperature, the amount of retained austenite increases and the ductility increases. But after reaching some maximum elongation, at still higher austempering temperature the stage II reaction becomes more pronounced and proceeds at a faster rate and the amount of retained austenite decreases at a faster rate than that at lower austempering temperature. Thus, this leads to some decrease of ductility.

4.3.3 Increase in UTS and YS of specimens with Cu over specimens without Cu:

4.3.3.1 Increase in UTS with austempering time and temperature:

Austempering Temp (°C)	Δ UTS (Mpa)			time(hr)	Δ UTS (Mpa)			
	time = 0.5 hr	time = 1 hr	time = 1.5 hr		T = 250°C	T = 300°C	T = 350°C	T = 400°C
250	38	45	40	0.5	38	48	62	68
300	48	52	56	1	45	52	60	49
350	62	60	60	1.5	40	56	60	50
400	68	49	50					

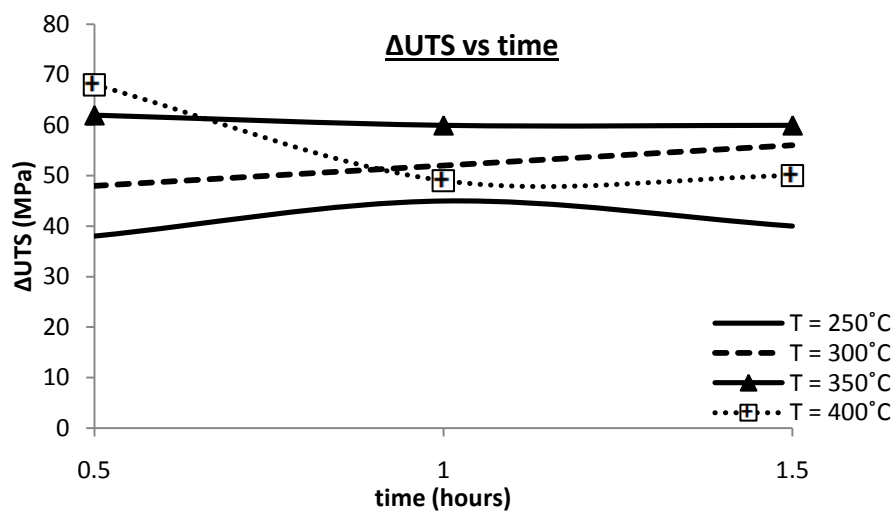
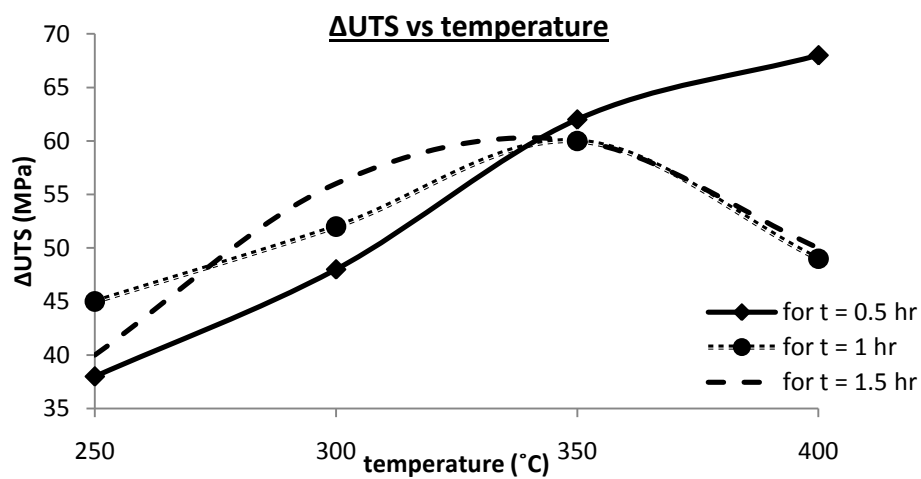


Fig 13. Increase in tensile strength of specimen with Cu in comparison to the specimen without Cu with a) temperature b) time

4.3.3.2 Increase in YS with austempering time and temperature:

Austempering Temp (°C)	ΔYS (Mpa)			ΔYS (Mpa)				
	time = 0.5 hr	time = 1 hr	time = 1.5 hr	time(hr)	T = 250°C	T = 300°C	T = 350°C	T = 400°C
250	30	37	30	0.5	30	50	68	72
300	50	46	36	1	37	46	73	71
350	68	73	69	1.5	30	36	69	79
400	72	71	79					

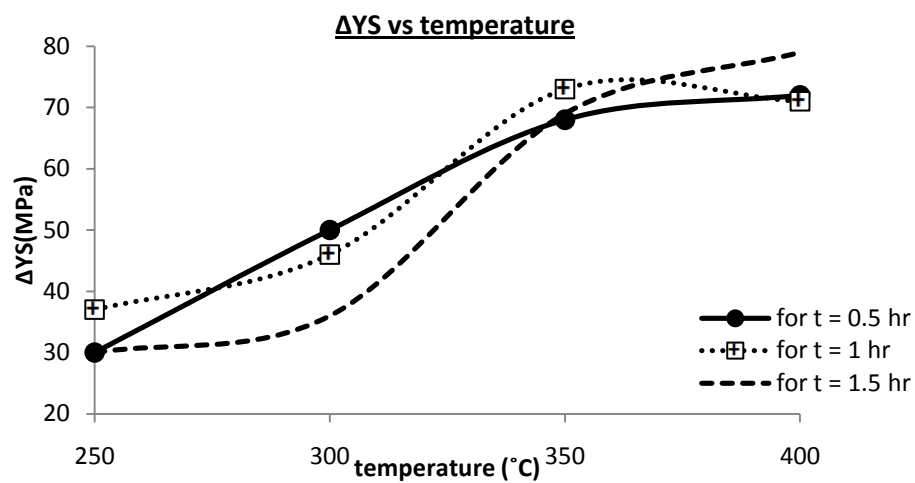


Fig 13(a)

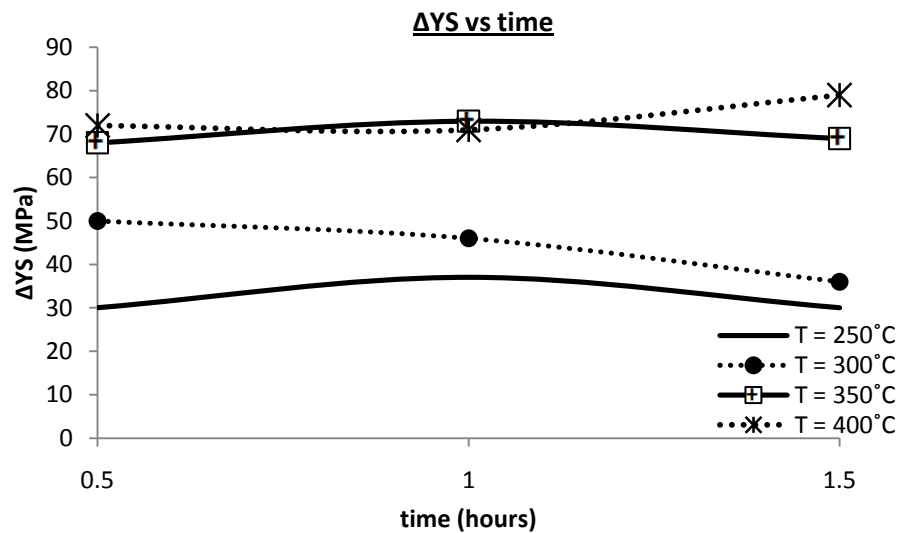


Fig 13 (b)

Fig 14. Increase in yield strength of specimen with Cu in comparison to the specimen without Cu with a) temperature b) time

4.3.3.3 Average increase in UTS and YS with austempering temperature:

Austempering Temp (°C)	avg increase in YS	avg increase in UTS
250	32	41
300	44	52
350	70	61
400	74	57

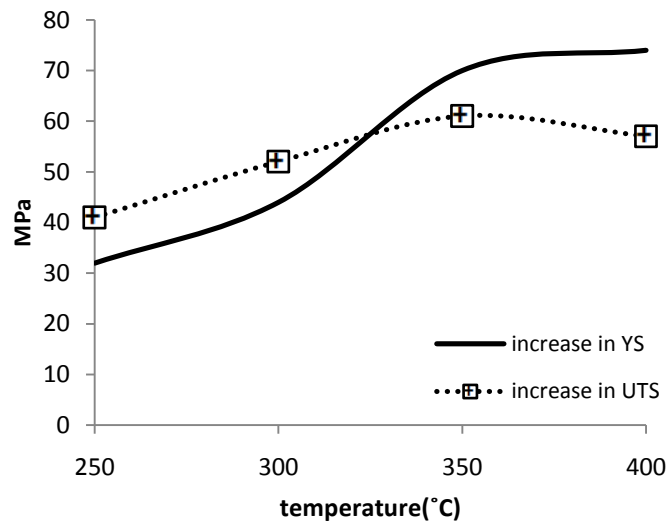
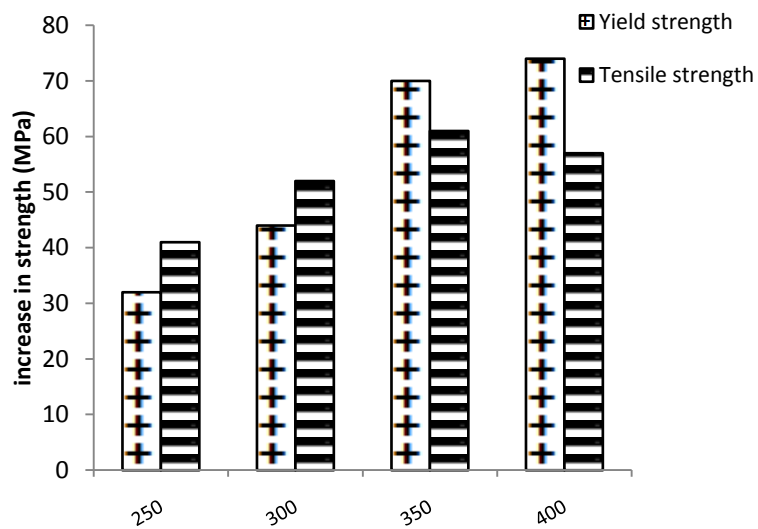


Fig 15. Average increase in strength with temperature of specimens alloyed with Cu in comparison to the specimens without Cu.

4.3.3.4 Discussion:

For specimens with Cu in comparison to the specimens without Cu, the rate of increase in yield strength and tensile strength is almost constant with time as shown in Fig 12 (b) and Fig 13 (b).

The rate of increase in yield strength and tensile strength for specimens with Cu in comparison to specimens without Cu, for different austempering times initially increases rapidly with temperature and then the rate of increase gradually becomes constant as shown in Fig 12 (a) and Fig 13 (a). In case of tensile strength, the rate of increase in strength decreases with temperature after reaching a certain peak value. From Fig 12(a), it is seen that, at austempering times above 1 hour, the rate of increase in tensile strength initially increases with temperature and reaches some peak value at 350°C and then starts decreasing with further rise of austempering temperature. At higher austempering temperatures, the effect of coarser bainitic ferrite in decreasing the strength may be more pronounced than the effect of pearlite matrix in increasing the strength of specimens alloyed with Cu. Hence, the rate of increase in strength for specimens alloyed with Cu in comparison to the specimens without Cu, decreases at high austempering temperatures

The average rate of increase in strength with temperature of specimen alloyed with Cu in comparison to the specimens without Cu is shown in Fig 14. There is a gradual increase in the rate of increase and then the increase reaches almost a constant value for higher austempering temperature.

CONCLUSIONS

1. Cu improves the overall mechanical properties of spheroidal graphite iron after austempering.
2. Cu increases the hardness, tensile strength and yield strength of austempered ductile iron.
3. The increase is constant with austempering time but with increasing austempering temperature it initially increases and then gradually became constant.
4. The ductility of austempered ductile iron is reduced by Cu.
5. The hardness, tensile strength and yield strength of ADI initially increases with austempering time and then after reaching certain maximum value they decreases.
6. The ductility of ADI also initially increases with austempering time up to a certain value and then it starts decreasing with further increase in time.
7. The hardness, tensile strength and yield strength of ADI decreases continuously with austempering temperature.
8. The ductility of ADI initially increases with austempering temperature and then after reaching some maximum value at around 350°C, it starts decreasing with further rise in temperature.

CHAPTER: 6

REFERENCES

1. J. Yang, S. K. Putatunda. Improvement in strength and toughness of austempered ductile cast iron by a novel two-step austempering process. *Mater. Des.* 25 (2004) 219 to 230
2. O. Eric, L. Sidjanin, Z. Miskovic, S. Zec, M.T. Jovanovic. Microstructure and toughness of Cu, Ni, Mo austempered ductile iron. *Mater. Lett.* 58 (2004) 2707 to 2711
3. S. K. Putatunda. Development of austempered ductile cast iron (ADI) with simultaneous high yield strength and fracture toughness by a novel two-step austempering process. *Mater. Sci. Eng., A* 315 (2001) 70 to 80
4. O. Eric, M. Jovanovic, L. Sidjanin, D. Rajnovic, S. Zec. The austempering study of alloyed ductile iron. *Mater. Des.* 27 (2006) 617–622
5. U. Batra, S. Ray, and S.R. Prabhakar. The influence of nickel and copper on austempering of ductile iron. *JMEPEG* (2004) 13:64-68
6. O. Eric, D. Rajnovic, L. Sidjanin, S. Zec, M. Jovanovic. An Austempering study of ductile iron alloyed with copper. *J. Serb. Chem. Soc.* 70 (7) (2005) 1015–1022
7. ASM Handbook Committee; Properties and selection, Iron and Steel, *Metals Handbook*, American Society for Metals , Metals Park, Ohio, Ninth Edition (1990)3-56
8. Ductile Iron society, ductile Iron Data for Design Engineers. <http://www.ductile.org/didata/pdf/didata2.pdf>,9
9. W. F. Smith; Principals of Materials Science and Engineering, Cast Irons, McGraw-Hill, Inc. Third Edition, 551-560
10. <http://www.ductile.org/didata/Section3/3part1.htm>
11. <http://www.ductile.org/didata/Section3/3part2.htm>
12. V. Singh. *Physical Metallurgy*. First edition (1999), Reprint 2007. Standard Publishers Distributers. 1705-B, Nai Sarak, New Delhi-110006.

13. S. K. Putatunda, I. Singh. Fracture toughness of unalloyed austempered ductile cast iron. J Test Eval 1995;23(5):325–32.
14. J. L. Doong, C. Chen. Fracture toughness of bainitic-nodular cast iron. Fatigue Fract Engg Mater Struct 1989;12:155–65.
15. D. J. Moore, T. N. Rouns, K. B. Rundamn. The effect of heat treatment, mechanical deformation and alloying elements on rate of bainitic formation in austempered ductile cast iron. J Heat Treat 1985;4(1):7–24.
16. J. R. Keogh; Section IV, Ductile Iron Society, (1998) 1-25
17. ADI Disadvantage. www.ctiparts.com/austempered.htm